SMITHSONIAN INSTITUTION

WASHINGTON 25, D. C.

February 13, 1968

Mr. John J. Ford, Jr.
176 Hendrickson Avenue
Rockville Centre
Long Island, New York

Dear Mr. Ford:

Dr. Stefanelli has asked me to comment on the portion of the Eric Newman report which deals with the X-ray diffraction examination of some USAOG gold pieces. The only reference to X-ray diffraction is contained in the first two sentences of the first full paragraph on page 9 of the report. It is quoted here for convenience:

> "Only coins #2,3,4,5,6 and 7 were tested by X-ray diffraction and the photographic prints have been interpreted to show that the planchets for coins #5 and 7 were cast rather than drawn while the others were not cast. The photographic results show a much greater dispersion of the rays passing through #5 and 7."

It is very difficult to interpret this report and know what the results of the X-ray diffraction tests actually are.

I would like to give you a short description of the fundamentals of X-ray diffraction and what is involved in a test of this type along with illustrations of the effects which I have prepared for this purpose.

A crystal, by definition, is a three dimensionally symmetric array of atoms or molecules in space. By virtue of its symmetry, a crystal will scatter X-rays at certain discrete angles according to the spacings between the atoms or molecules in the crystal, and the wavelength of the X-rays. This scattering gives rise to the concept of planes existing in the crystal. X-ray diffraction follows the Bragg Law $n\lambda = 2d \sin\theta$ where λ is the wavelength of the X-rays, d is the spacing between the planes and θ is the angle at which diffraction occurs.

A photographically recorded X-ray diffraction diagram (back-reflection "Laue" pattern) of a single crystal consists of a pattern

of dots, images of the incident X-ray beam as it is diffracted in the crystal lattice. The position of a particular spot on the pattern is characteristic of certain parallel lattice planes and their d spacing, which diffracted the incident beam. If we have more than one single crystal, as the number of differently oriented crystals in the incident beam increases the number of spots on the film increases and the spots from a particular series of parallel lattice planes effectively merge to form a ring on the photographic film

When a single crystal is permanently strained the spots on the diagram become elongated due to local differences in the lattice spacings.

A piece of metal such as a coin, unless specially treated, is an aggregate of many small crystals, each oriented differently and therefore gives rise to a series of concentric rings in the Laue pattern.

When a piece of metal is reshaped mechanically, such as by striking a coin, the crystals become irregularly distorted and the spacing of the lattice planes is changed. This change in spacing gives rise to broadened, diffuse diffraction rings on a photographic film.

When the same piece of metal is heated, i.e. annealed, the crystals are relaxed and reassume their characteristic lattice spacing. The diffraction rings become sharp and well-defined. If annealed at too high a temperature the metal will recrystallize, grain growth will occur and the diffraction rings will become spotty due to the decreased number of crystals in the incident beam.

For the purpose of illustrating the effects very briefly described above, I took a portion of an 1844 U. S. $5 gold piece which had no numismatic value and melted it and cast a small irregularly shaped button, Fig. 2. I made a back reflection X-ray diffraction photograph of the as-cast structure, Fig. 1. This diffraction diagram contains a relatively small number of spots making this a diagram which indicates large grain size. The discrete and undistorted character of each individual spot indicates an unstrained lattice.

I next placed the button of 900 fine gold in a hydraulic press and compressed it at approximately 40,000 pounds per square inch to produce the cold worked button shown in Fig. 4. The diffraction diagram shown in Fig. 3 of this cold worked structure shows a broadened diffuse diffraction ring due to the distortion of the crystalline structure and multiplication of the number of crystals.

I next annealed the button overnight at approximately 600° C. Recrystallization and some grain growth occurred. The diffraction diagram of this recrystallized structure is shown in Fig. 6.

Figure 5 is a diffraction diagram of a portion of the coin as received. It shows the broadened diffuse diffraction ring which is characteristic of a cold worked structure.

It is clear from the foregoing that X-ray back-reflection photographs can yield patterns characteristic of the condition of a metal sample viz: (1) Spotty diffraction in general indicates either a cast structure or a recrystallized structure; (2) Broad diffuse rings can be indicative of a cold worked distorted structure. Perhaps this will put you into a position to reach some tentative conclusions on your own about the patterns discussed in the Newman report if you have copies of them in your possession.

In the Newman report the second sentence summarizes his results. I cannot say, without seeing the diffraction diagrams what is meant by dispersion in this context.

In the context of crystallography, dispersion is generally used in conjunction with light optical methods and is the measure of the change of the principal indices of refraction with frequency. In X-ray diffraction analysis the dispersion effect has to do with anomalies in the atomic scattering factor. An adequate discussion of this is beyond the scope of this letter and I will refer you to the book <u>X-ray Diffraction</u>, A. Gunier, W. H. Freeman and Company, San Francisco, 1963. I feel that the word dispersion is not properly used in the context of the Newman report.

The second point I would like to comment on is the statement, "...the rays passing through..." The X-rays do penetrate into the sample, are diffracted, and emerge through that same surface in back reflection technique. In this sense the X-rays do pass through the fabric of the sample, but do not traverse the coin in the sense of entering the obverse and emerge from the reverse.

I am very sorry that I don't have time to prepare more illustrative material and give you a more complete discussion of the problem of examining metallic sample by the back reflection X-ray diffraction technique. An excellent text on this subject is <u>X-ray Metallography</u>, by A. Taylor, Wiley & Sons, New York, 1961. This book is fairly complete and runs to almost 1000 pages.

Due to the brevity of this particular discussion and the necessarily incomplete technical details, I request that you do not publish any portion of this letter without my prior permission.

Incidentially, I just received the call for papers for the annual Denver Conference on Applications of X-ray Analysis and the technical emphasis this year is to be placed on X-ray Diffraction Metallography. The conference is to be held August 21-23 in Estes Park, Colorado. The final program will probably not be announced

until about the first of June. If you want more information you can write to:

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Sincerely yours,

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cc: Dr. V. Clain-Stefanelli

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Figure 1 Back-reflection X-ray diffraction diagram of 900 fine gold button as-cast

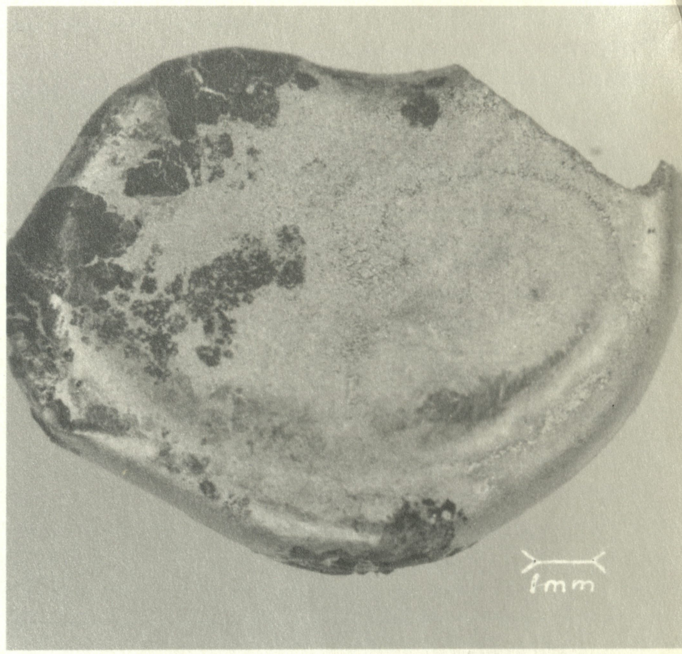
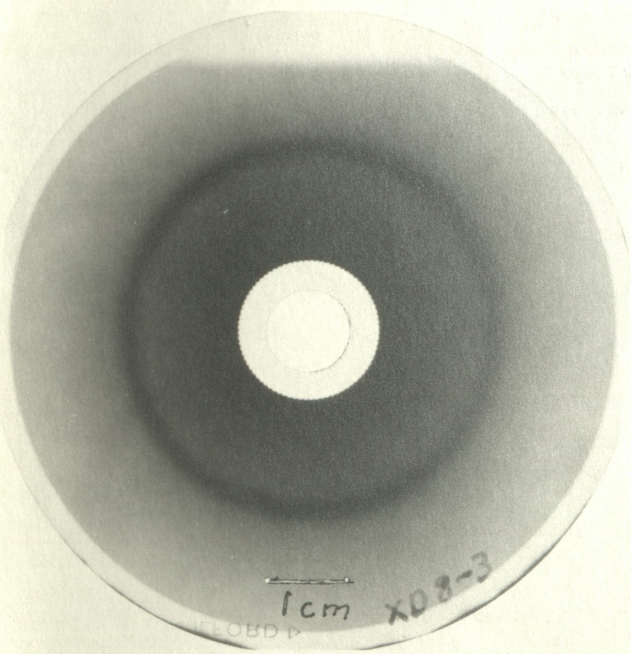


Fig.2 Button 900 fine gold as cast



ure 3 Back-reflection X-ray diffraction diagram of 900 fine gold button as cold worked

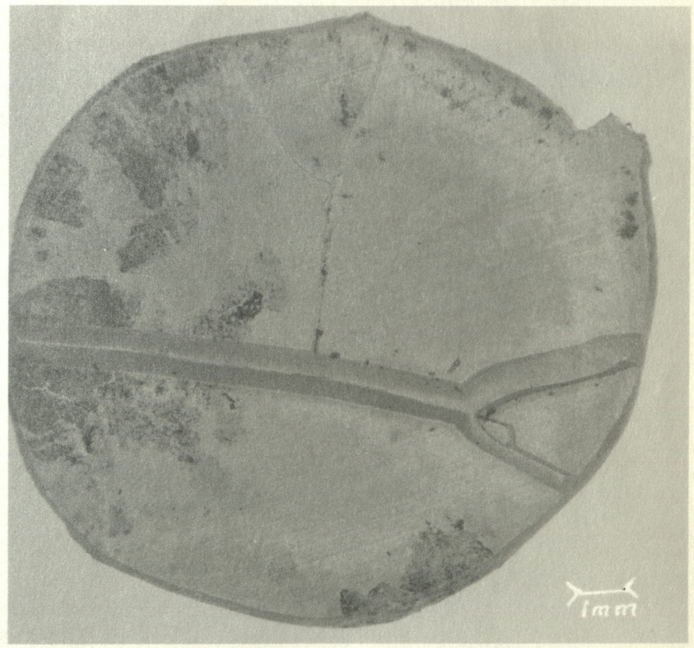


Figure 4 Button 900 fine gold as cold worked and annealed

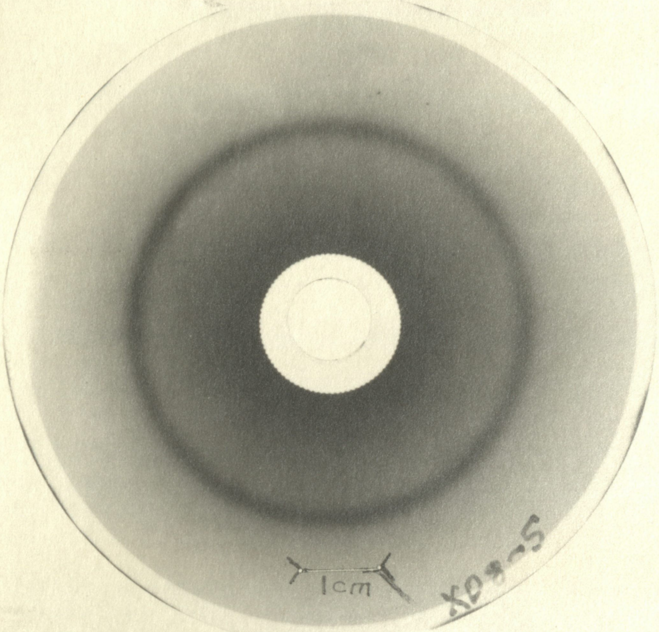


Figure 5 Back reflection X-ray diffraction diagram of 1844 U. S. $5 gold piece as received.

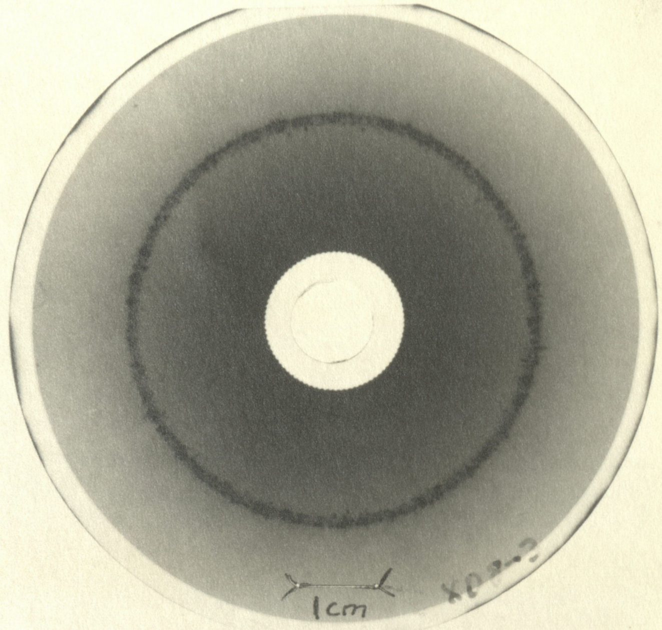


Figure 6 Back reflection X-ray diffraction diagram of 900 fine gold button after heat treatment.